

# EVALUATION OF CLEANING PROCESSES

## UTILIZING RADIOISOTOPES

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INSTRUMENTATION AND NUCLEAR STUDIES SECTION

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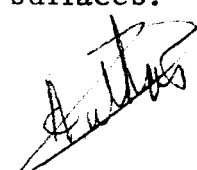
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## SUMMARY

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What are the merits of different processes used to clean surfaces? This problem was examined at Litton and resolved by using radioisotopes to contaminate the specimens under test. A minute quantity of radioisotope was deposited on the surface of the test specimens; the parts were measured for radioactivity before and after the cleaning process using a Geiger-Muller detector. The ultrasonic cleaning processes evaluated consisted of a cascade series of numerous cleaning solutions or baths such as Tergitol, Trend, or Turco No. 3878 preceded by a Chlorothene Nu degreaser step. It was found that the Turco bath was the most effective cleaning solution in comparison to the Tergitol, Trend, and Chlorothene Nu. The optimum cleaning time was 2 minutes for the Turco, 3 minutes for the Tergitol bath, 4 minutes for the Trend bath, and 15 minutes for the Chlorothene Nu.

The technique of applying a radioisotope to the surface parts resulted in an effective method of determining surface cleanliness; however, unless adequate precautions are taken, the cleaning efficiency data secured may not represent the actual operational conditions and may instead represent an evaluation of processes for decontamination of radioactive surfaces.



## SECTION 1

### INTRODUCTION

#### 1-1. GENERAL

Emphasis on surface cleanliness accentuates the proper selection of cleaning processes and surface contamination detection techniques. Techniques for determining surface cleanliness for vital parts of electro-mechanical systems, such as gyro components, require greater accuracy than ever before with the improved cleaning methods now being utilized in industry. One approach to this problem is to determine cleaning efficiency using radioactive isotopes to dope the test specimens. This method was utilized at Litton. Prior to subjecting the gyro components to a typical cleaning process, small quantities of radioactive isotopes (radioisotopes) were applied to the surface of the test specimen. The surface cleanliness was determined by comparing the amount of radioactivity before and after the cleaning process. However, this comparison does not always represent the actual effectiveness of the cleaning process, as is discussed in Section 2-3.

Tests for surface cleanliness of gyro components were performed after cleaning the components with a degreaser step followed by a series of ultrasonic bath immersions. The radioisotopes used to determine the effectiveness of removing surface contamination consisted of the following solutions:

- a. Phosphorous-32 water-base inorganic sodium di-hydrogen phosphate ( $\text{NaH}_2\text{PO}_4$ ) solution
- b. Phosphorous-32 oil-miscible solution
- c. Iodine-131 water-base solution (sodium iodide buffered with sulfite)
- d. Iodine-131 oil-base solution completely organic bound.

A single radioactive isotope was applied in a thin coat to selected portions of a gyro center case and a gyro micro balance frame. These devices are shown in Figure 1. On the center case, the radioisotope was applied to its internal annular depression. On the micro balance frame, the isotope was applied in its center dot depression. In the few cases where the planchet was used, the radioisotope was applied on the bottom inside surface. The amount of radioactivity deposited on the part surface, expressed in terms

of disintegrations per second, was approximately 500 dps. The instruments used to measure the radioisotope consisted of a Geiger-Muller Detector (Nuclear Chicago D37A) shielded by a Technical Associates Model LS-6 lead shield. After application of one of the radioisotopes, each part was then placed separately into the G-M detector and measured for activity. The parts were positioned individually in such a manner that the identical conditions could be obtained for the measurements after cleaning.

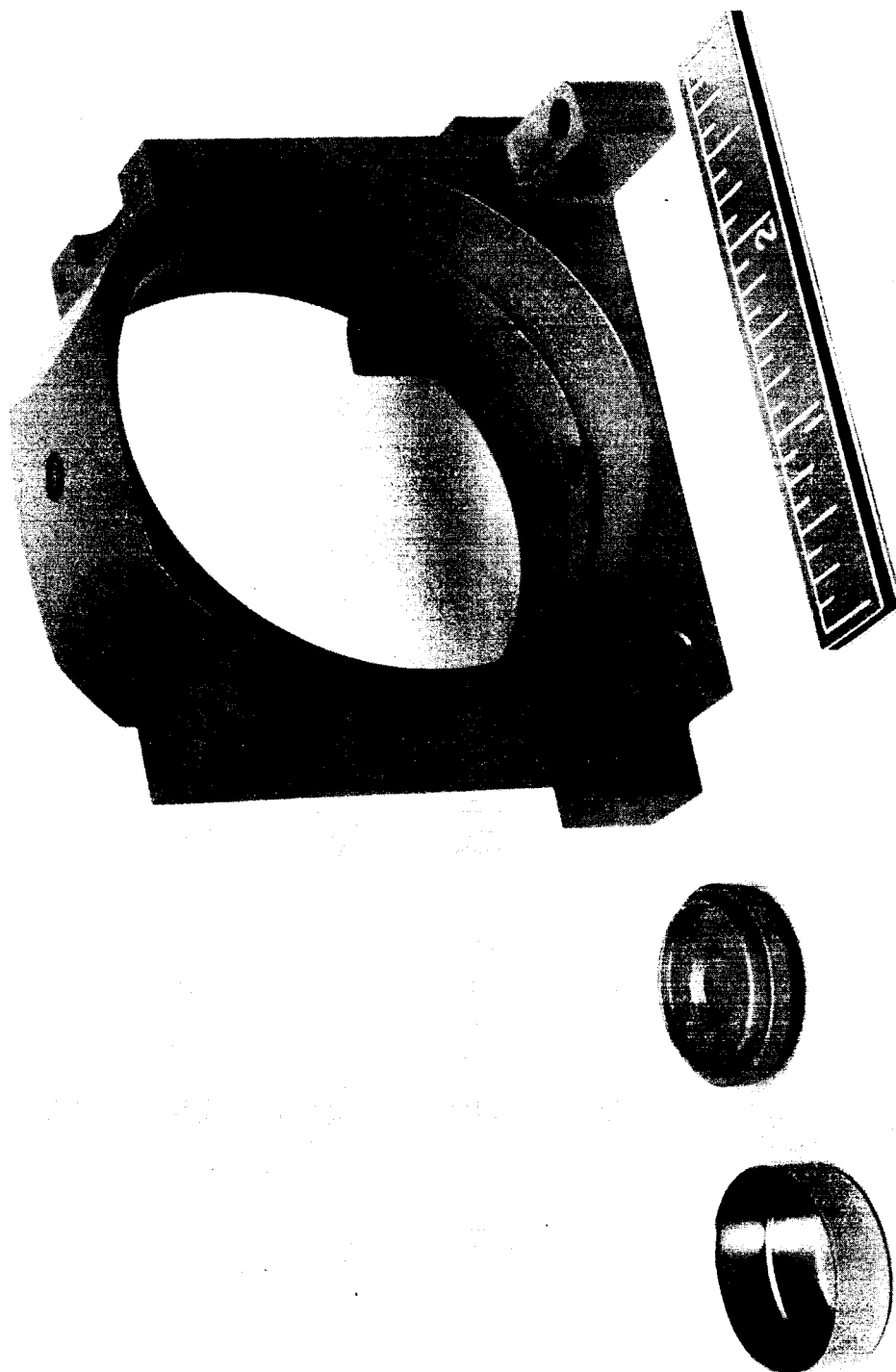


Figure 1. Gyro Components & Planchet

## SECTION 2

### CLEANING PROCESS EVALUATION

#### 2-1. TEST METHOD

Small amounts of radioisotope were deposited on gyro component test specimens. After the activity of the radioisotope was measured, the specimens were subjected to either a 5 or 7 step cleaning process which is outlined briefly below:

Step 1. Parts placed in Chlorothene Nu vapor degreaser tank for approximately 15 minutes. Then the parts were raised to the top of the tank (above the vapor line) and allowed to air dry.

Step 2. Parts were placed in ultrasonic cleaner containing Trend solution for 2 minutes. The Trend solution was a 1- to 2-percent solution maintained at a minimum temperature of 140°F.

Step 3. Parts rinsed immediately after removing from Trend solution with deionized water.

Step 4. Parts placed in Tergitol ultrasonic cleaner solution for 2 to 3 minutes. The Tergitol solution was a 1- to 2-percent solution maintained at a minimum temperature of 140°F.

Step 5. Parts rinsed again with deionized water before placing in 212°F dryer for 20 to 25 minutes. With the 7 step process, drying is not employed as a part of step 5.

Step 6. Parts placed in ultrasonic cleaner containing Turco No. 3878 solution. This bath consists of a solution of 20 percent Turco No. 3878 and 80 percent water, maintained at a minimum temperature of 140°.

Step 7. Parts rinsed with deionized water before placing in 212°F dryer for 20 to 25 minutes.

After completion of the cleaning process, the specimens were again placed in the detector chamber to measure the remaining radioactivity. The cleaning efficiency was determined by comparing the radioactive counts before and after the cleaning process. Although the half-life of the radioisotopes was much longer than the time between pre- and post-measurements, this parameter was not neglected.



TABLE 1  
CLEANING EFFICIENCY FOR FIVE STEP PROCESS

Radio-isotope	Mixture	Specimen Tested	Cleaning Efficiency* (%)
I-131	Oil Base	Center Case	93.5
	Oil Base	Center Case	96.8
	Oil Base	Micro Balance Frame	96.4
	Oil Base	Micro Balance Frame	98.8
I-131	Water Base	Micro Balance Frame	93.9
	Water Base	Micro Balance Frame	92.0
P-32	Water Base	Center Case	31.0
	Water Base	Center Case	12.2
	Water Base	Micro Balance Frame	5.0
	Water Base	Micro Balance Frame	0.7
P-32	Oil Miscible (no oil added)	Micro Balance Frame	47.0
	Oil Miscible (no oil added)	Micro Balance Frame	47.8
	Oil Miscible (no oil added)	Stainless-Steel Planchet	98.8
	Oil Miscible (no oil added)	Stainless-Steel Planchet	>99.5
	Oil Miscible (50% solution olive oil)	Micro Balance Frame	46.6
	Oil Miscible (50% solution olive oil)	Micro Balance Frame	49.8
	Oil Miscible (50% solution olive oil)	Stainless-Steel Planchet	99.3
	Oil Miscible (50% solution olive oil)	Stainless-Steel Planchet	>99.5

\*Percent removal of radioactivity.

NOTE: Micro Balance Frame and Center Case made of aluminum material, anodized.

TABLE 2  
CLEANING EFFICIENCY FOR SEVEN STEP PROCESS

Sample	Radioisotope	Specimen Tested	Cleaning Efficiency* (%)
1	I-131 (oil base)	Micro Balance Frame	89.0
2	I-131 (oil base)	Micro Balance Frame	98.2
3	P-32 (water base)	Micro Balance Frame	<0.5**
4	P-32 (water base)	Micro Balance Frame	<0.5

\*Percent removal of radioactivity

\*\*Accuracy limit is approximately 0.5 percent by count.

## 2-2. PROCESS EVALUATION

The effectiveness of the 5 and 7 step cleaning process with the three test specimens (Figure 1) are shown in Tables 1 and 2. After the overall cleaning efficiency was determined, as shown in these tables, the effectiveness of each process step (5 step series) was ascertained for two of the radioisotopes. In this phase of the test, two micro balance frame parts were deposited with the oil-base I-131 and two with the water-base P-32 radioisotope contaminant. After each part was individually checked for radioactivity, the parts first were subjected to Step 1 of the cleaning procedure (Chlorothene Nu vapor degreaser bath) for 15 minutes. After completion of the vapor degreaser bath, radioactivity of the parts was measured again before subjecting the parts to the 2-minute ultrasonic Trend bath and the deionized water rinse (Steps 2 and 3 of cleaning procedure). Again the radioactivity of the parts was measured in the G-M detector chamber. The parts were then placed in the ultrasonic Tergitol bath for 2 minutes, rinsed with deionized water, and oven dried. The final contamination radioactivity measurement was then taken. The results are summarized graphically in Figure 2. These results show that the Chlorothene Nu removed approximately 61 percent of the oil base I-131 and less than 0.5 percent of the water base P32. As can also be noted from Figure 2, there is a slight scatter of data resulting from different positioning of the test samples in the cleaning bath. The cleaning efficiency for the complete 5 step process averaged approximately 94 percent for both oil-base I-131 samples, and 2.5 percent for both water-base P-32 specimens. This apparent discrepancy in the determination of cleaning efficiency led to the use of the steel planchet test specimen and to investigation of the role of the radioisotope on cleaning efficiency measurements as discussed in Section 2-3.

## 2-3. RADIOISOTOPE EVALUATION

The radioisotope surface contaminants utilized in the first series of tests consisted of water-base P-32 and oil-base I-131. The cleaning efficiency using the reverse base isotopes (water-base I-131 and oil-miscible P-32 surface contaminants) were also investigated. By reversing the form of each radioisotope solution, the effectiveness of removing the surface contaminant was tested as a function of the isotope carrier or base material.

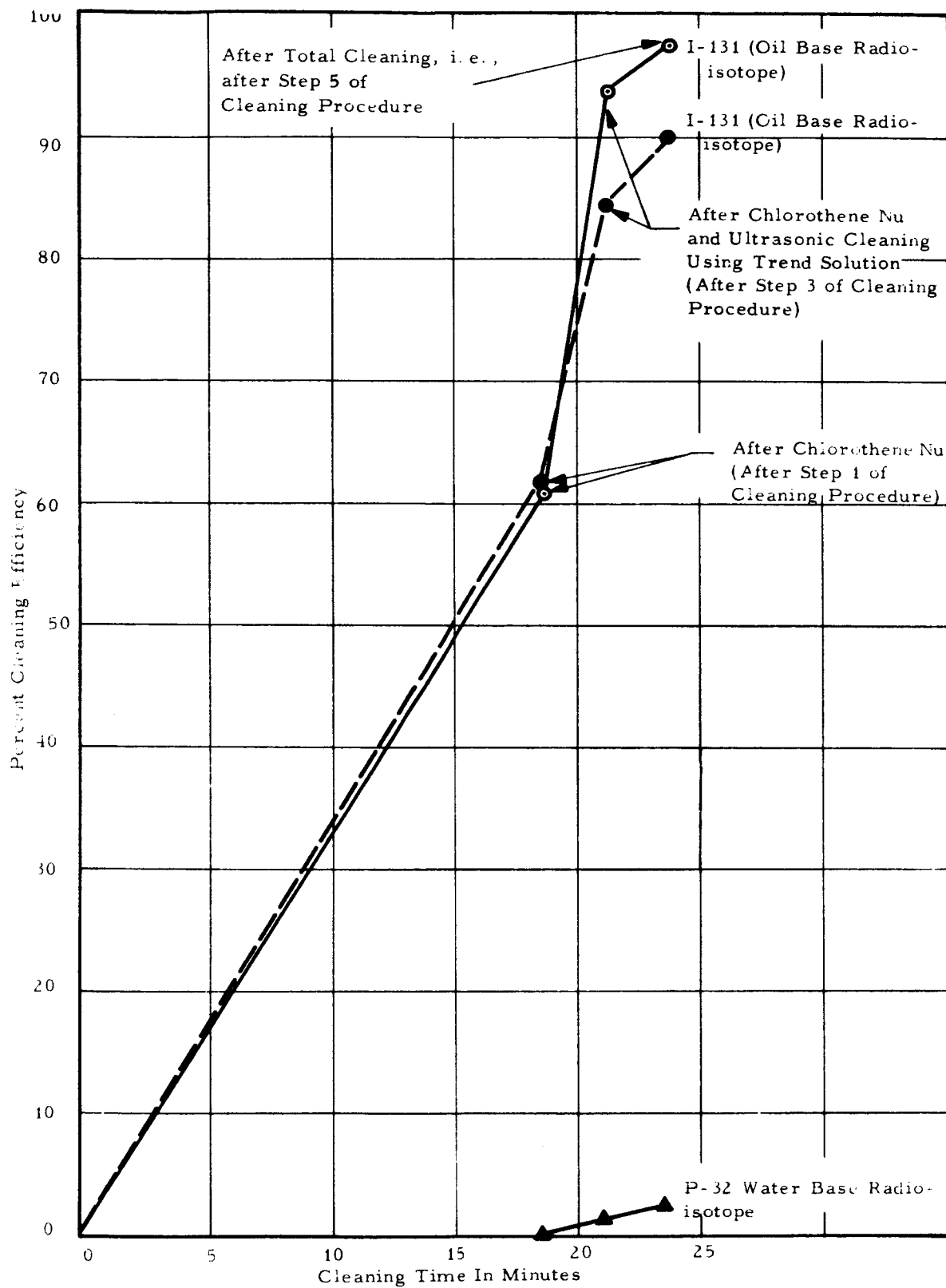


Figure 2. Cleaning Efficiency for Five Step Process

In the case of the water-base I-131 radioisotope contaminant, the cleaning efficiency using the five step cleaning process was slightly less than that of the oil-base I-131. This difference could be caused by the surface tension of the oil, it being much higher than that of water, thus preventing the adsorption of the oil-base I-131 into the metal pores. Another reason for the difference is the possible experimental errors of approximately 5 percent resulting from slight variation of the cleaning solution and/or the positioning of the test specimen into the cleaning baths. The high cleaning efficiency when using the I-131 in either form indicates that the ultrasonic cleaning process is effective in removing contaminants. The secured data in this case does represent the actual cleaning efficiency of the processes.

The previous experiments, using the water base-P-32 radioisotope, indicated that the cleaning efficiency for both the five and seven step process was very poor. By substituting the oil-miscible P-32 radioisotope as the surface contaminant of the micro balance frame test specimen, the cleaning efficiency using the five step process increased from less than 5 percent to approximately 47 percent.

When the oil-miscible P-32 (no oil added) radioisotope solution was deposited on the micro balance frame, the cleaning efficiency for the five step process did not exceed 50 percent. Further experiments were conducted using stainless-steel planchets as test specimens in order to resolve the low cleaning efficiency. By substituting the stainless-steel planchet in place of the micro balance frame as the test specimen, the cleaning efficiency increased to greater than 98 percent, as shown in Table 1. It was thus determined that the P-32 reacted with the aluminum, with some sort of mechanism thus preventing proper cleaning. In addition, it was found that as an oil content was added to the oil-miscible P-32 radioisotope (20- to 80-percent solution), the cleaning efficiency of the process (with the micro balance frame specimen) increased approximately an additional 15 percent.

### SECTION 3

#### EVALUATION OF EACH PROCESS STEP VS TIME

##### 3-1. STEP-BY-STEP EVALUATION

This section provides an evaluation of each cleaning process step versus time. Unless otherwise stated, each step is applicable to both the five and seven step processes. In addition it should be noted that each specimen was freshly doped prior to its test, i. e. , no test was made using the residual radioactivity from a prior test.

Step 1. Chlorothene Nu Degreaser. - The Chlorothene Nu degreaser is a vapor degreasing tank where parts are maintained for a 10- to 15-minute period. When the parts are placed into the Chlorothene Nu vapor bath, the vapor condenses on the surface of the parts, resulting in a cleaning effect as the condensed vapor drips from the parts.

The test to determine the cleaning efficiency as a function of cleaning time was accomplished by subjecting the parts to 5, 10, 15, and 25 minutes cleaning time while taking radioactivity measurements after each time increment. The results of this test show the optimum cleaning time for the oil base solution was 15 minutes. The results of this test and the following tests of this section are presented graphically in Figure 3. The water-base P-32 solution was not tested in the Chlorothene Nu in this manner because previous tests indicated that the degreaser was completely ineffective against this radioisotope.

Steps 2 and 3. Ultrasonic Cleaner - Trend (Bath) Solution. - The ultrasonic cleaning bath, which contains Trend solution, normally is scheduled for 2 or 3 minutes in the cleaning cycle. A test was conducted to find the cleaning efficiency as a function of time. The parts were contaminated with an oily film containing I-131 radioisotope or an inorganic sodium dihydrogen phosphate doped with water-base P-32 radioisotope. Each specimen was subjected to 1, 2, 4, 6, and 10 minutes cleaning time in the ultrasonic-Trend bath, rinsed and its radioactivity measured. The results show that a cleaning time of 4 minutes was required to reduce the oil-base I-131 surface contamination to less than 8 percent. When the parts are subjected to less than 4 minutes cleaning time, the cleaning efficiency is marginal.

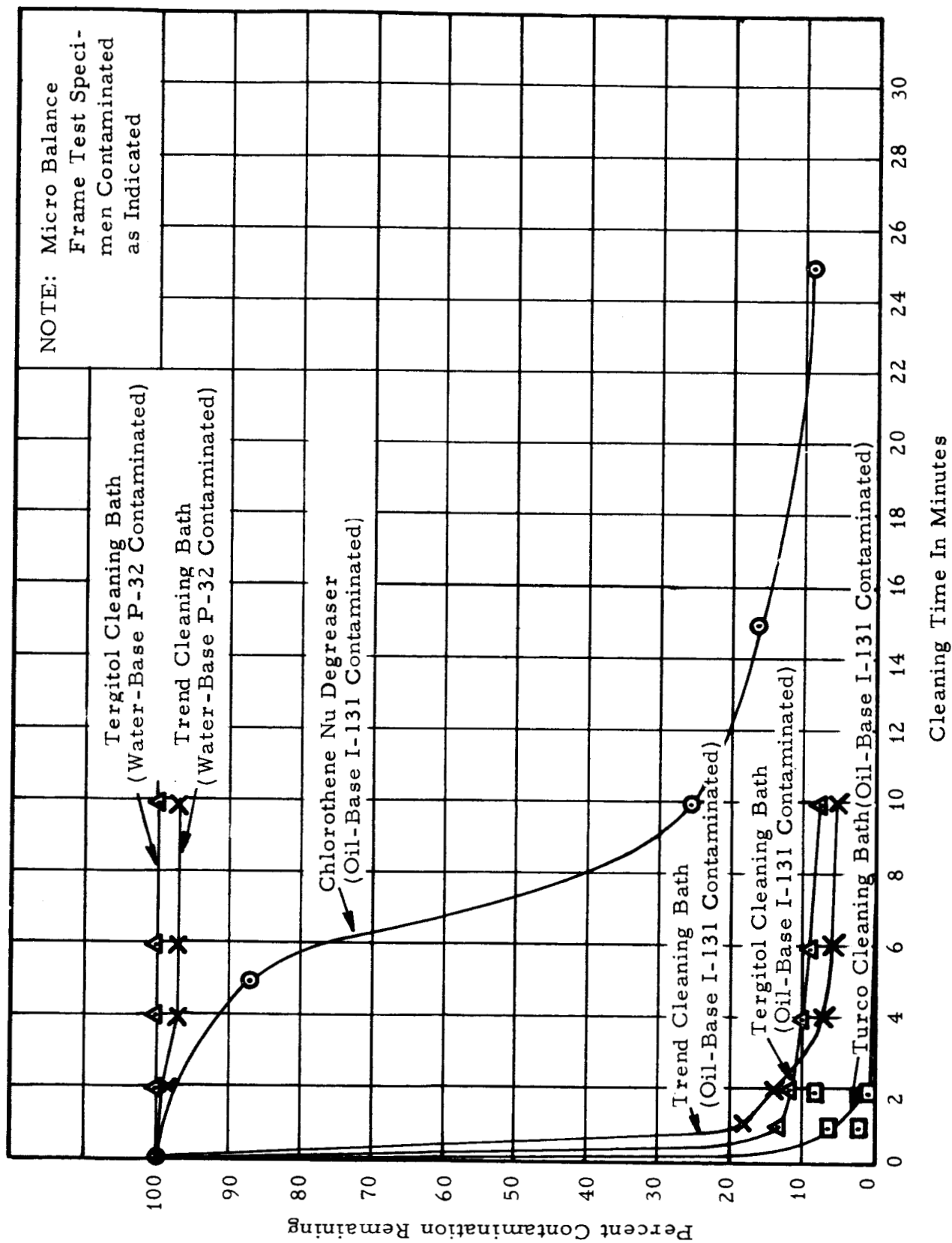


Figure 3. Percent Contamination Remaining for Individual Cleaning Steps

It should be noted that Figure 3 shows the relationship of percent contamination remaining versus cleaning time rather than the cleaning efficiency versus cleaning time. It can be seen that the Trend cleaning bath is effective during the first 4 minutes; after the first 4 minutes, its cleaning effectiveness is insignificant. As for the water-base P-32 contaminant, the data indicates inadequate cleaning results as would be suspected.

Steps 4 and 5. Ultrasonic-Cleaner, Tergitol (Bath) Solution. - The ultrasonic cleaning bath containing Tergitol solution was tested in the same manner as the Trend solution, and the optimum cleaning time for the oil base I-131 solution was 2 to 3 minutes. The initial cleaning for Tergitol was somewhat better than for Trend; however, the final cleaning (>4 minutes) shows that Trend surpasses Tergitol because of less surface contamination remaining on the part.

Steps 6 and 7. Ultrasonic Cleaner With Turco Solution - The ultrasonic Turco cleaner test was conducted using a cleaning time of 1, 2, 3, and 4 minutes. The Turco bath proved extremely effective for removing the oil-base I-131 contaminate. As can be seen from Table 4 and Figure 3, an optimum cleaning time of 2 minutes was found. There was a slight scatter of data for the Turco bath caused by positioning the parts at different locations in the bath. The percent residual contamination remaining on the parts was approximately 1 percent after 2 minutes and less than 0.5 percent after 3 minutes cleaning time. This means that the Turco cleaning action surpasses both that of Trend and Tergitol. The P-32 test specimens were not included in this test, as prior tests (Table 2) shows that the complete seven step process is ineffective for cleaning against this radioisotope.

The use of the Turco solution is limited by its possible corrosive action on some materials. This aspect should not be overlooked. The Turco manufacturer specifies the compatibility of the No. 3878 solution with various materials.

It should be noted that the cleaning efficiency of the ultrasonic baths is higher than the Chlorothene Nu degreaser step and that these baths require much less process time. It should be ascertained through further research or evaluation if the "degreaser" step in the cleaning process could be eliminated inasmuch as considerable savings in time and money could be visualized.